



# Environmental Technology Verification Report

## Explosives Detection Technology

### Research International, Inc. FAST 2000™



Environmental Security  
Technology Certification  
Program

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Oak Ridge National Laboratory

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**THE ENVIRONMENTAL TECHNOLOGY VERIFICATION  
PROGRAM**

**ETV** ✓



U.S. Environmental Protection Agency



Environmental Security  
Technology Certification  
Program



Oak Ridge National Laboratory

## Joint Verification Statement

<b>TECHNOLOGY TYPE:</b>	<b>EXPLOSIVES DETECTION</b>	
<b>APPLICATION:</b>	<b>MEASUREMENT OF EXPLOSIVES IN CONTAMINATED WATER</b>	
<b>TECHNOLOGY NAME:</b>	<b>FAST 2000™</b>	
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The U.S. Environmental Protection Agency (EPA) has created the Environmental Technology Verification Program (ETV) to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high quality, peer reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized standards and testing organizations and stakeholder groups consisting of regulators, buyers, and vendor organizations, with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

The Department of Defense (DoD) has a similar verification program known as the Environmental Security Technology Certification Program (ESTCP). The purpose of ESTCP is to demonstrate and validate the most promising innovative technologies that target DoD's most urgent environmental needs and are projected to

pay back the investment within 5 years through cost savings and improved efficiencies. ESTCP demonstrations are typically conducted under operational field conditions at DoD facilities. The demonstrations are intended to generate supporting cost and performance data for acceptance or validation of the technology. The goal is to transition mature environmental science and technology projects through the demonstration/ validation phase, enabling promising technologies to receive regulatory and end user acceptance in order to be fielded and commercialized more rapidly.

The Oak Ridge National Laboratory (ORNL) is one of the verification organizations operating under the Site Characterization and Monitoring Technologies (SCMT) program. SCMT, which is administered by EPA's National Exposure Research Laboratory, is one of 12 technology areas under ETV. In this demonstration, ORNL evaluated the performance of explosives detection technologies. This verification statement provides a summary of the test results for Research International's (RI's) FAST 2000™. This verification was conducted jointly with the Department of Defense's (DoD's) Environmental Security Technology Certification Program (ESTCP).

### **DEMONSTRATION DESCRIPTION**

This demonstration was designed to evaluate technologies that detect and measure explosives in soil and water. RI elected to analyze only water samples with the FAST 2000. The demonstration was conducted at ORNL in Oak Ridge, Tennessee, from August 23 through September 1, 1999. Spiked samples of known concentration were used to assess the accuracy of the technology. Explosives-contaminated water samples from Tennessee, Oregon, and Louisiana with concentrations ranging from 0 to 25,000 µg/L were analyzed. The primary constituents in the samples were 2,4,6-trinitrotoluene (TNT); isomeric dinitrotoluene (DNT), including both 2,4-dinitrotoluene and 2,6-dinitrotoluene; hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX); and octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX). The results of the water analyses conducted under field conditions by the FAST 2000 were compared with results from reference laboratory analyses of homogenous replicate samples determined using EPA SW-846 Method 8330. Details of the demonstration, including a data summary and discussion of results, may be found in the report entitled *Environmental Technology Verification Report: Explosives Detection Technology—Research International, Inc., FAST 2000™*, EPA/600-R-00/045.

### **TECHNOLOGY DESCRIPTION**

The FAST 2000 is based on a displacement assay that uses antibodies and fluorescence as a means of detection. The unit (weighing 2.8 lb, with dimensions of 6 × 15.5 × 16 cm) can be easily carried into the field and plugged directly into a portable PC for on-site data acquisition and analysis. The key elements of the sensor are (1) antibodies specific for the analyte; (2) signal molecules that are similar to the analyte but are labeled with a fluorophore (a cyanine-based fluorescent dye, Cy5) to enable fluorescence detection; and (3) a fluorescence detector. For analysis, the analyte-specific antibodies are immobilized onto a solid support and then saturated with the fluorescently labeled signal molecule, creating an antibody/signal molecule complex. Monoclonal antibodies (the Naval Research Laboratory's 11B3 TNT and Strategic Diagnostics RDX) are immobilized onto porous membrane supports and saturated with the fluorescent tag. The membrane is inserted into a disposable coupon and placed in the FAST 2000, and the buffer flow is started by a computer command. Once the fluorescence background signal due to unbound Cy5 has stabilized (generally 15–20 minutes), the biosensor is ready for sample injection. If the sample contains the target analyte, a proportional amount of the labeled signal molecule is displaced from the antibody and detected by the fluorimeter downstream. The coupon and membrane can be used for repeated assays. The life of the membrane is dependent upon the number and concentration of positive assays that are run. The reporting limit for both TNT and RDX was 20 µg/L.

## VERIFICATION OF PERFORMANCE

The following performance characteristics of the FAST 2000 were observed:

**Precision:** For the water samples, the mean relative standard deviations (RSDs) for RDX and TNT were 52% and 76%, respectively.

**Accuracy:** The mean recoveries for RDX and TNT were 192% and 316%, respectively.

**False positive/false negative results:** Of the 20 blank water samples, RI reported RDX in 4 samples (24% false positives) and TNT in 16 samples (80% false positives). Three of the RDX results were reported as “ME,” which indicated that the sample had “matrix effects” and the result could not be reported by the FAST 2000. False positive and false negative results were also determined by comparing the FAST 2000 result to the reference laboratory result on environmental and spiked samples (e.g., whether the FAST 2000 reports a result as a nondetect that the reference laboratory reported as a detect, and vice versa). For RDX, 2% of the results were false positives relative to the reference laboratory result, while 16% of the TNT results were reported as false positives. RI reported a small fraction of the samples (3% for each analyte) as nondetects (i.e., false negatives) when the laboratory reported a detect.

**Completeness:** Approximately 80% of the water analyses were complete. Approximately 18% of the RDX results and 21% of the TNT results were reported as “matrix effects,” where a result could not be obtained.

**Comparability:** A one-to-one sample comparison of the FAST 2000 results and the reference laboratory results was performed for all samples (spiked and environmental) that were reported above the reporting limits. The correlation coefficient ( $r$ ) for the comparison of the entire water data set for TNT was 0.23, and the slope ( $m$ ) of the linear regression line was 1.81. When comparability was assessed for specific concentration ranges, the  $r$  value did not change dramatically for TNT, ranging from 0.14 to 0.21 depending on the concentration ranges selected. RDX correlation with the reference laboratory for water was higher ( $r = 0.63$ ,  $m = 1.60$ ). Examination of the data indicated that the RDX results were usually higher than those of the reference laboratory. However, for specific environmental sample matrices (such as the samples from the Louisiana Army Ammunition Plant), the FAST 2000 results were generally lower than those of the reference laboratory. This indicated the possibility of a matrix-dependent effect.

**Sample Throughput:** Operating under the outdoor conditions, the RI team, usually consisting of three operators, accomplished a sample throughput rate of approximately three samples per hour for the water analyses. Separate instruments were used for the TNT and RDX analyses. Typically, two operators analyzed samples while one operator performed data analysis, but the technology can be run by a single trained operator.

**Overall Evaluation:** The verification team found that the FAST 2000 was relatively simple for the trained analyst to operate in the field, requiring less than an hour for initial setup. The overall performance of the FAST 2000 for the analysis of water samples was characterized as imprecise and biased high for TNT, and imprecise and biased high (but matrix-dependent) for RDX. As with any technology selection, the user must determine if this technology is appropriate for the application and the project data quality objectives. For more information on this and other verified technologies, visit the ETV web site at <http://www.epa.gov/etv>.

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**NOTICE:** EPA and ESTCP verifications are based on evaluations of technology performance under specific, predetermined criteria and appropriate quality assurance procedures. EPA, ESTCP, and ORNL make no expressed or implied warranties as to the performance of the technology and do not certify that a technology will always operate as verified. The end user is solely responsible for complying with any and all applicable federal, state, and local requirements. Mention of commercial product names does not imply endorsement or recommendation.

# **Environmental Technology Verification Report**

## **Explosives Detection Technology**

### **Research International, Inc. FAST 2000™**

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Environmental Security Technology Certification Program



## **Notice**

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## Abbreviations and Acronyms

2-Am-DNT	2-amino-4,6-dinitrotoluene, CAS # 35572-78-2
4-Am-DNT	4-amino-2,6-dinitrotoluene, CAS # 1946-51-0
CFI	Continuous Flow Immunosensor
CRREL	U.S. Army Cold Regions Research and Engineering Laboratory
2,4-DNT	2,4-dinitrotoluene, CAS # 121-14-2
2,6-DNT	2,6-dinitrotoluene, CAS # 606-20-2
DNT	isomeric dinitrotoluene (includes both 2,4-DNT and 2,6-DNT)
DoD	U.S. Department of Defense
EPA	U.S. Environmental Protection Agency
ERA	Environmental Resource Associates
ESTCP	Environmental Security Technology Certification Program
ETV	Environmental Technology Verification Program
fn	false negative result
fp	false positive result
GC	gas chromatography
HMX	octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine, CAS # 2691-41-0
HPLC	high-performance liquid chromatography
IMS	ion mobility spectrometry
LAAAP	Louisiana Army Ammunition Plant
ME	matrix effects
MLAAP	Milan Army Ammunition Plant
NERL	National Exposure Research Laboratory (EPA)
NRL	U.S. Naval Research Laboratory
ORNL	Oak Ridge National Laboratory
PE	performance evaluation sample
QA	quality assurance
QC	quality control
RDX	hexahydro-1,3,5-trinitro-1,3,5-triazine, CAS # 121-82-4
RI	Research International, Inc.
RSD	relative standard deviation
SCMT	Site Characterization and Monitoring Technologies Pilot of ETV
SD	standard deviation
TNB	1,3,5-trinitrobenzene, CAS # 99-35-4
TNT	2,4,6-trinitrotoluene, CAS # 118-96-7



## Section 1 — Introduction

The U.S. Environmental Protection Agency (EPA) created the Environmental Technology Verification Program (ETV) to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized standards and testing organizations and stakeholder groups consisting of regulators, buyers, and vendor organizations, with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing verification test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance (QA) protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

ETV is a voluntary program that seeks to provide objective performance information to all of the participants in the environmental marketplace and to assist them in making informed technology decisions. ETV does not rank technologies or compare their performance, label or list technologies as acceptable or unacceptable, seek to determine “best available technology,” or approve or disapprove technologies. The program does not evaluate technologies at the bench or pilot scale and does not conduct or support research. Rather, it conducts and reports on testing designed to describe the performance of technologies under a range of environmental conditions and matrices.

The program now operates 12 pilots covering a broad range of environmental areas. ETV has begun with a 5-year pilot phase (1995–2000) to test a wide range of partner and procedural alternatives in various pilot areas, as well as the true market demand for and response to such a program. In these pilots, EPA utilizes the expertise of partner “verification organizations” to design efficient processes for conducting performance tests of innovative technologies. These expert partners are both public and private organizations, including federal laboratories, states, industry consortia, and private sector entities. Verification organizations oversee and report verification activities based on testing and QA protocols developed with input from all major stakeholder/customer groups associated with the technology area. The verification described in this report was administered by the Site Characterization and Monitoring Technologies (SCMT) Pilot, with Oak Ridge National Laboratory (ORNL) serving as the verification organization. (To learn more about ETV, visit ETV’s Web site at <http://www.epa.gov/etv>.) The SCMT pilot is administered by EPA’s National Exposure Research Laboratory (NERL), Environmental Sciences Division, in Las Vegas, Nevada.

The Department of Defense (DoD) has a similar verification program known as the Environmental Security Technology Certification Program (ESTCP). The purpose of ESTCP is to demonstrate and validate the most promising innovative technologies that target DoD’s most urgent environmental needs and are projected to pay back the investment within 5 years through cost savings and improved efficiencies. ESTCP responds to (1) concern over the slow pace and cost of remediation of environmentally contaminated sites on military installations, (2) congressional direction to conduct demonstrations specifically focused on new technologies, (3) Executive Order 12856, which requires federal agencies to place high priority on obtaining funding and resources needed for the development of innovative pollution prevention programs and technologies for installations and in acquisitions, and (4) the need to improve defense

readiness by reducing the drain on the Department's operation and maintenance dollars caused by real world commitments such as environmental restoration and waste management. ESTCP demonstrations are typically conducted under operational field conditions at DoD facilities. The demonstrations are intended to generate supporting cost and performance data for acceptance or validation of the technology. The goal is to transition mature environmental science and technology projects through the demonstration/ validation phase, enabling promising technologies to receive regulatory and end user acceptance in order to be fielded and commercialized more rapidly. (To learn more about ESTCP, visit ESTCP's web site at <http://www.estcp.org>.)

EPA's ETV program and DoD's ESTCP program established a memorandum of agreement in 1999 to work cooperatively with ESTCP on the verification of technologies that are used to improve environmental cleanup and protection at both DOD and non-DOD sites. The verification of field analytical technologies for explosives detection described in this report was conducted jointly by ETV's SCMT pilot and ESTCP. The verification was conducted at ORNL in Oak Ridge, Tennessee, from August 23 through September 1, 1999. The

performances of two field analytical techniques for explosives were determined under field conditions. Each technology was independently evaluated by comparing field analysis results with those obtained using an approved reference method, EPA SW-846 Method 8330. The verification was designed to evaluate the field technology's ability to detect and measure explosives in soil and water. The primary constituents in the samples were 2,4,6-trinitrotoluene (TNT); isomeric dinitrotoluene (DNT), including both 2,4-dinitrotoluene (2,4-DNT) and 2,6-dinitrotoluene (2,6-DNT); hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX); and octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX). Naturally contaminated environmental soil samples, ranging in concentration from 0 to approximately 90,000 mg/kg, were collected from DoD sites in California, Louisiana, Iowa, and Tennessee, and were used to assess several performance characteristics. Explosives-contaminated water samples from Tennessee, Oregon, and Louisiana with concentrations ranging from 0 to 25,000 µg/L were also evaluated. This report discusses the performance of the Research International, Inc., FAST 2000™ instrument for the analysis of water samples only. Research International elected not to analyze the soil samples.

## Section 2 — Technology Description

*In this section, the vendor (with minimal editorial changes by ORNL) provides a description of the technology and the analytical procedure used during the verification testing activities.*

### General Technology Description

The Continuous Flow Immunosensor (CFI) is based on a displacement assay that utilizes antibodies and fluorescence as a means of detection. The technology was originally developed by the U.S. Naval Research Laboratory (NRL). The field-portable version of the CFI, the FAST 2000, has been engineered and manufactured by Research International, Inc. (RI). The FAST 2000 unit (shown in Figure 1) can be easily carried into the field (weight: 2.8 lb; dimensions: 6 × 15.5 × 16 cm) and plugged directly into a portable PC for on-site data acquisition and analysis.



**Figure 1.** The FAST 2000.

The key elements of the sensor are (1) antibodies specific for the analyte; (2) signal molecules that are similar to the analyte but are labeled with a fluorophore (a cyanine-based fluorescent dye, Cy5) to enable fluorescence detection; and (3) a fluorescence detector. For analysis, the analyte-specific antibodies are immobilized onto a solid support and then saturated with the fluorescently labeled signal molecule, creating an antibody/signal molecule complex. Monoclonal antibodies (the Naval Research Laboratory's 11B3 TNT and Strategic Diagnostics RDX) are immobilized onto porous membrane supports and

saturated with the fluorescent tag using the detailed protocols outlined in draft U.S. EPA Method 4655. The membrane is inserted into a disposable coupon and placed in the FAST 2000, and the buffer flow is started by a computer command. Once the fluorescence background signal due to unbound Cy5 has stabilized (generally 15–20 min), the biosensor is ready for sample injection. If the sample contains the target analyte, a proportional amount of the labeled signal molecule is displaced from the antibody and detected by the fluorimeter downstream. The coupon and membrane can be used for repeated assays. The life of the membrane is dependent upon the number and concentration of positive assays that are run.

At the time of the demonstration, the cost of purchasing the FAST 2000 was \$23,650. Instrument purchase included the FAST 2000 instrument designed for use with an immunoassay-based sensor; a data acquisition card and a cable linking the instrument to the laptop computer; a fluid storage unit; one assay coupon kit; the software required to run the instrument and analyze data; and an instruction manual. This price did not include the cost of the laptop. The FAST 2000 could also be leased for \$1970 per month.

### Preparation of Standards

The TNT and RDX calibration standards were prepared by drying down 20  $\mu\text{L}$  of stock explosive standard (1,000,000  $\mu\text{g/L}$  stored in acetonitrile) with a nitrogen air stream. Using a micropipettor, 2.0 mL of system flow buffer (10 mM sodium monophosphate, 2.5% ethanol, and 0.01% Tween, pH 7.4) was added to the tube to dissolve the explosive residue, forming a 10,000- $\mu\text{g/L}$  explosive standard. Serial dilutions of the 10,000- $\mu\text{g/L}$  standard were made in flow buffer to obtain 25-, 50-, 100-, 250-, 500-, and 1,000- $\mu\text{g/L}$  standards.

### Sample Preparation and Analysis

Sample preparation was minimal. Briefly, 40  $\mu\text{L}$  of 0.5 M sodium phosphate/0.5% Tween 20 and 50  $\mu\text{L}$  ethanol were added to 1.91 mL of water sample. Samples and standards (150  $\mu\text{L}$ ) were injected into the FAST 2000 using a 1- $\mu\text{L}$  Hamilton gas-tight syringe. Sample analyses using the FAST 2000 immunosensor were initiated with an injection of the 500- $\mu\text{g}/\text{L}$  explosive standard. After duplicate injections of a water sample were analyzed, a second standard was analyzed. The concentration of the second standard was based on the response determined from the sample. The analyst confirmed the computer-calculated peak area, which corresponded to the start of the peak and the end of the peak, as

designated by the analyst. The peak area of the closest standard was then compared to the peak area from each sample injection to acquire a concentration for that injection of the sample. The calculated concentrations from the duplicate sample injections were averaged to determine the final result for the sample. The reporting limit for both TNT and RDX was 20  $\mu\text{g}/\text{L}$ .

### Cross-Reactivity

Table 1 contains a list of compounds that may interfere with the analyses because they are known to cross-react with the TNT or RDX assay. Approximate levels of cross-reactivity, in terms of relative response to the antibody, are provided in the table.

**Table 1.** FAST 2000 Cross-reactivity

Compound	Anti-RDX antibody cross-reactivity (%)	Anti-TNT antibody cross-reactivity <sup>a</sup> (%)	Compound	Anti-RDX antibody cross-reactivity (%)	Anti-TNT antibody cross-reactivity <sup>a</sup> (%)
RDX	100	1	Tetryl	0.95	38
TNT	1.8	100	2,4-Dinitrotoluene	3.1	20
HMX	4.8	5	2,6-Dinitrotoluene	1.1	4
2-Nitrotoluene	1.9	9	Trinitrolycerin	1.4	ND
3-Nitrotoluene	2.6	ND	2-Amino-4,6-dinitrotoluene	1.3	21
4-nitrotoluene	3.0	ND	4-Amino-2,6-dinitrotoluene	1.8	1
Nitrobenzene	1.9	16	1,2-Dinitrolycerin	1.8	ND
1,3-Dinitrobenzene	2.8	ND	1,3-Dinitrolycerin	1.3	ND
1,3,5-Trinitrobenzene	3.8	600	Dinitroethylene glycol	1.9	ND

<sup>a</sup>ND = not determined.

## Section 3 — Demonstration Design

### Objective

The purpose of this section is to describe the demonstration design. It is a summary of the technology demonstration plan (ORNL 1999).

### Demonstration Testing Location and Conditions

The verification of field analytical technologies for explosives was conducted at the ORNL Freels Bend Cabin site, in Oak Ridge, Tennessee. The site is somewhat primitive, with no running water, but the vendors were provided with some shelter (porch overhang) and electrical power. The temperature and relative humidity were monitored during field testing. Over the ten days of testing, the average temperature was 77°F, and ranged from 60 to 88°F. The average relative humidity was 67%, and ranged from 35 to 96%.

The samples used in this study were brought to the demonstration testing location for evaluation by the vendors. Explosives-contaminated soils from Army ammunition plants in Iowa, Louisiana, and Tennessee and a former Army base in California (Fort Ord) were used in this verification. In addition, explosives-contaminated water samples were analyzed from DoD sites in Oregon, Louisiana, and Tennessee. Because samples were obtained from multiple DoD sites, the samples represented a reasonable cross section of the population of explosives-contaminated matrices, such that the versatility of the field technology could be evaluated. The vendors had the choice of analyzing either soil or water samples, or both matrices. More specific details about the samples are presented below.

### Soil Sample Descriptions

The primary constituents in the soil samples were TNT, DNT, RDX, and HMX. The samples also contained trace amounts of 2-amino-4,6-dinitrotoluene (2-Am-DNT) and 4-amino-2,6-dinitrotoluene (4-Am-DNT), which are degradation products of TNT. The total concentration of explosives ranged from 0 to approximately 90,000 mg/kg. The following sections describe the sites from which the samples were collected.

### Sources of Samples

#### Iowa Army Ammunition Plant

Currently an active site, the Iowa Army Ammunition Plant was constructed to load, assemble, and pack various conventional ammunition and fusing systems. Current production includes 120-mm tank rounds, warheads for missiles, and mine systems. During the early years of use, the installation used surface impoundments, landfills, and sumps for disposal of industrial wastes containing explosives. The major contaminants in these samples were TNT, RDX, and HMX.

#### Louisiana Army Ammunition Plant

The Louisiana Army Ammunition Plant (LAAAP), near Shreveport, Louisiana, is a government-owned facility that began production in 1942. The facility is currently an Army Reserve plant. Production items at LAAAP have included metal parts for artillery shells; the plant also loads, assembles, and packs artillery shells, mines, rockets, mortar rounds, and demolition blocks. As a result of these activities and the resulting soil and groundwater contamination, EPA placed LAAAP on the National Priorities List of contaminated sites (Superfund) in 1989. The major constituents in the samples from this site were TNT, RDX, and HMX, with trace levels of 1,3,5-trinitrobenzene (TNB), DNT, 2-Am-DNT, and 4-Am-DNT.

#### Milan Army Ammunition Plant

Currently active, the Milan Army Ammunition Plant (MLAAP) in Milan, Tennessee, was established in late 1940 as part of the pre-World War II buildup. The facility still has ten ammunition loading, assembly, and packaging lines. Munitions-related wastes have resulted in soil contamination. The primary contaminants in these soils were RDX and TNT.

#### Volunteer Army Ammunition Plant

The Volunteer Army Ammunition Plant, in Chattanooga, Tennessee, was built in 1941 to manufacture TNT and DNT. All production ceased in 1977. Past production practices resulted in significant soil and groundwater contamination. In the samples from this site, concentrations of TNT

and DNT ranged from 10 to 90,000 mg/kg, with significantly smaller concentrations of Am-DNT isomers.

### **Fort Ord Military Base**

Fort Ord, located near Marina, California, was opened in 1917 as a training and staging facility for infantry troops and was closed as a military installation in 1993. Since then, several nonmilitary uses have been established on the site: California State University at Monterey Bay has opened its doors on former Fort Ord property, the University of California at Santa Cruz has established a new research center there, the Monterey Institute of International Studies will take over the officer's club and several other buildings, and the post's airfield was turned over to the city of Marina. The Army still occupies several buildings.

An Army study conducted in 1994 revealed that the impact areas at the inland firing ranges of Fort Ord were contaminated with residues of high explosives (Jenkins, Walsh, and Thorne 1998). Fort Ord is on the National Priorities List of contaminated sites (Superfund), requiring the installation to be characterized and remediated to a condition that does not pose unacceptable risks to public health or the environment. The contaminant present at the highest concentration (as much as 300 mg/kg) was HMX; much lower concentrations of RDX, TNT, 2-Am-DNT, and 4-Am-DNT are present.

### **Performance Evaluation Samples**

Spiked soil samples were obtained from Environmental Resource Associates (ERA, Arvada, Colo.). The soil was prepared using ERA's semivolatile blank soil matrix. This matrix was a 40% clay topsoil that had been dried, sieved, and homogenized. Particle size was 60 mesh and smaller. The samples, also referred to as performance evaluation (PE) samples, contained known levels of TNT and RDX. The concentrations that were evaluated contained 10, 50, 100, 250, and 500 mg/kg of each analyte. Prior to the demonstration, ORNL analyzed the spiked samples to confirm the concentrations. The method used was a modified Method 8330, similar to the reference

laboratory method described in Section 4. For the demonstration, four replicates were prepared at each concentration level.

Blank soil samples were evaluated to determine the technology's ability to identify samples with no contamination (i.e., to ascertain the false positive error rate). The soil was collected in Monroe County, Tennessee, and was certified by ORNL to be free of contamination prior to verification testing. A reasonable number of blanks ( $N = 20$ ) was chosen to balance the uncertainty for estimating the false positive error rate and the required number of blank samples to be measured.

### **Soil Sample Preparation**

A few weeks prior to the demonstration, all of the soil samples were shipped in plastic Ziplock bags at ambient temperature to ORNL. The samples were stored frozen ( $<0^{\circ}\text{C}$ ) prior to preparation. To ensure that the developers and the reference laboratory analyzed comparable samples, the soils were homogenized prior to sample splitting. The process was as follows. The sample was kneaded in the Ziplock bag to break up large clumps. Approximately 1500 g of soil was poured into a Pyrex pan, and debris was removed. The sample was then air-dried overnight. The sample was sieved using a 10-mesh (2-mm particle size) screen and placed in a 1-L widemouthed jar. After thorough mixing with a metal spatula, the sample was quartered. After mixing each quarter, approximately 250 g from each quarter was placed back in the 1-L widemouthed jar, for a total sample amount of approximately 1000 g. Analysis by the ORNL method confirmed sample homogeneity (variability of 20% relative standard deviation or less for replicate measurements). The sample was then split into subsamples for analysis during the demonstration. Each 4-oz sample jar contained approximately 20 g of soil. Four replicate splits of each soil sample were prepared for each participant. The design included a one-to-one pairing of the replicates, such that the vendor and reference lab samples could be directly matched. Three replicate sets of samples were also prepared for archival storage. To ensure that degradation did not occur, the soil samples were frozen ( $<0^{\circ}\text{C}$ ) until analysis (Maskarinec et al. 1991).

## **Water Sample Descriptions**

### ***Sources of Samples***

Explosives-contaminated water samples from Tennessee, Oregon, and Louisiana were analyzed. The contamination in the water samples ranged in concentration from 0 to about 25,000 µg/L. Water samples were collected from LAAAP, MLAAP, and Volunteer, described in the previous section (see “Sources of Samples”). Water samples were also obtained from Umatilla Chemical Depot, described below.

Umatilla Chemical Depot is located in northeastern Oregon. The mission of the facility recently changed to storage of chemical warfare ammunition. Once the chemicals are destroyed, the installation is scheduled to close. Several environmental sites have been identified for cleanup prior to base closure. One site has explosives-contaminated groundwater; the cleanup identified for this site is to pump and treat the water with granulated activated carbon. The major contaminants in these samples were TNT, RDX, HMX, and TNB. According to a remedial investigation conducted at the site, these samples were not contaminated with any chemical warfare agents.

### ***Performance Evaluation Samples***

Water samples of known concentration were prepared by the U.S. Army Cold Regions Research and Engineering Laboratory (CRREL) in Hanover, New Hampshire. These samples were used to determine the technology’s accuracy. The concentrations of TNT and RDX in the spiked distilled water samples were 25, 100, 200, 500, and 1000 µg/L for each analyte; four replicates were prepared at each concentration. Prior to the demonstration, ORNL analyzed the spiked samples to confirm the concentrations.

Distilled water obtained from ORNL was used for the blanks. As with the soil samples, 20 blank samples were analyzed.

### ***Water Sample Preparation***

The water samples were collected in 2.5-gal carboys approximately 7 to 10 days prior to the start of the demonstration and shipped on ice to ORNL. To ensure that degradation did not occur, the samples

were stored under refrigeration until analysis (~4°C) (Maskarinec et al. 1999). Sample splitting was performed in a small laboratory cold room, which was maintained at 4°C. To prepare the water sample, a spout was attached to the 2.5-gal carboy, and the water sample was split by filling multiple 250-mL amber glass bottles. As with the soil samples, four replicate splits of each water sample were prepared for each participant, and three sets of samples were also prepared for archival storage.

### **Sample Randomization**

The samples were randomized in two stages. First, the order in which the filled jars were distributed was randomized so that the same developer did not always receive the first jar filled for a given sample set. Second, the order of analysis was randomized so that each participant analyzed the same set of samples, but in a different order. Each jar was labeled with a sample number. Replicate samples were assigned unique (but not sequential) sample numbers. Spiked materials and blanks were labeled in the same manner, such that these quality control samples were indistinguishable from other samples. All samples were analyzed blindly by both the developer and the reference laboratory.

### **Summary of Experimental Design**

The distribution of samples from the various sites is described in Table 2. A total of 108 soil samples were analyzed, with approximately 60% of the samples being naturally contaminated environmental soils, and the remaining 40% being spikes and blanks. A total of 176 water samples were analyzed, with approximately 75% of the samples being naturally contaminated environmental water, and the remaining 25% being spikes and blanks. Four replicates were analyzed for each sample type. For example, four replicate splits of each of three Fort Ord soils were analyzed, for a total of 12 individual Fort Ord samples.

### **Description of Performance Factors**

In Section 5, technology performance is evaluated in terms of precision, accuracy, completeness, and comparability, which are indicators of data quality (EPA 1998). False positive and negative results, sample throughput, and ease of use are also

evaluated. Each of these performance characteristics is defined in this section.

**Table 2.**Summary of Soil and Water Samples

Sample source or type	No. of soil samples	No. of water samples
Fort Ord	12	0
Iowa	4	0
LAAAP	16	80
MLAAP	20	20
Umatilla	0	24
Volunteer	12	8
Spiked	24	24
Blank	20	20
Total	108	176

### **Precision**

Precision is the reproducibility of measurements under a given set of conditions. Standard deviation (SD) and relative standard deviation (RSD) for replicate results are used to assess precision, using the following equation:

$$RSD = (SD / \text{average concentration}) \times 100\% . \quad (\text{Eq. 1})$$

The overall RSD is characterized by three summary values:

- mean — i.e., average;
- median — i.e., 50th percentile value, at which 50% of all individual RSD values are below and 50% are above; and
- range — i.e., the highest and lowest RSD values that were reported.

The average RSD may not be the best representation of precision, but it is reported for convenient reference. RSDs greater than 100% should be viewed as indicators of large variability and possibly non-normal distributions.

### **Accuracy**

Accuracy represents the closeness of the technology's measured concentrations to known (in this case, spiked/PE) values. Accuracy is assessed in terms of percent recovery, calculated by the following equation:

$$\% \text{ recovery} = (\text{measured concentration} / \text{known concentration}) \times 100\% . \quad (\text{Eq. 2})$$

As with precision, the overall percent recovery is characterized by three summary values: mean, median, and range.

### **False Positive/Negative Results**

A false positive (fp) result is one in which the technology detects explosives in the sample when there actually are none (Berger, McCarty, and Smith 1996). A false negative (fn) result is one in which the technology indicates that no explosives are present in the sample, when there actually are (Berger, McCarty, and Smith 1996). The evaluation of fp and fn results is influenced by the actual concentration in the sample and includes an assessment of the reporting limits of the technology. False positive results are assessed in two ways. First, the results are assessed relative to the blanks (i.e., the technology reports a detected value when the sample is a blank). Second, the results are assessed on environmental and spiked samples where the analyte was not detected by the reference laboratory (i.e., the reference laboratory reports a nondetect and the field technology reports a detection). False negative results, also assessed for environmental and spiked samples, indicate the frequency that the technology reported a nondetect (i.e., < reporting limits) and the reference laboratory reported a detection. Note that the reference laboratory results were confirmed by the ORNL laboratory so that fp/fn assessment would not be influenced by faulty laboratory data. The reporting limit is considered in the evaluation. For example, if the reference laboratory reported a result as 0.9 mg/kg, and the technology's paired result was reported as below reporting limits (<1 mg/kg), the technology's result was considered correct and not a false negative result.

### **Completeness**

Completeness is defined as the percentage of measurements that are judged to be usable (i.e., the result is not rejected). The acceptable completeness is 95% or greater.

### **Comparability**

Comparability refers to how well the field technology and reference laboratory data agree. The difference between accuracy and comparability is that whereas accuracy is judged relative to a known value, comparability is judged relative to the results of a standard or reference procedure, which may or may not report the results accurately. A one-to-one sample comparison of the technology results and the reference laboratory results is performed in Section 5.

A correlation coefficient quantifies the linear relationship between two measurements (Draper and Smith 1981). The correlation coefficient is denoted by the letter  $r$ ; its value ranges from  $-1$  to  $+1$ , where  $0$  indicates the absence of any linear relationship. The value  $r = -1$  indicates a perfect negative linear relation (one measurement decreases as the second measurement increases); the value  $r = +1$  indicates a perfect positive linear relation (one measurement increases as the second measurement increases). The slope of the linear regression line, denoted by the letter  $m$ , is related to  $r$ . Whereas  $r$  represents the linear association between the vendor and reference laboratory concentrations,  $m$  quantifies the amount of change in the vendor's measurements relative to the reference laboratory's measurements. A value of  $+1$  for the slope indicates perfect agreement. Values greater than  $1$  indicate that the vendor results are generally higher than the reference laboratory, while values less than  $1$  indicate that the vendor results are usually lower than the reference laboratory. In addition, a direct comparison between the field technology and reference laboratory data is performed by evaluating the percent difference (%D) between the measured concentrations, defined as

$$\%D = ([field\ technology] - [ref\ lab]) / (ref\ lab) \times 100\% \quad (\text{Eq. 3})$$

The range of %D values is summarized and reported in Section 5.

### **Sample Throughput**

Sample throughput is a measure of the number of samples that can be processed and reported by a technology in a given period of time. This is reported in Section 5 as number of samples per hour times the number of analysts.

### **Ease of Use**

A significant factor in purchasing an instrument or a test kit is how easy the technology is to use. Several factors are evaluated and reported on in Section 5:

- What is the required operator skill level (e.g., technician, B.S., M.S., or Ph.D.)?
- How many operators were used during the demonstration? Could the technology be run by a single person?
- How much training would be required in order to run this technology?
- How much subjective decision-making is required?

### **Cost**

An important factor in the consideration of whether to purchase a technology is cost. Costs involved with operating the technology and the standard reference analyses are estimated in Section 5. To account for the variability in cost data and assumptions, the economic analysis is presented as a list of cost elements and a range of costs for sample analysis. Several factors affect the cost of analysis. Where possible, these factors are addressed so that decision makers can independently complete a site-specific economic analysis to suit their needs.

### **Miscellaneous Factors**

Any other information that might be useful to a person who is considering purchasing the technology is documented in Section 5. Examples of information that might be useful to a prospective purchaser are the amount of hazardous waste generated during the analyses, the ruggedness of the technology, the amount of electrical or battery power necessary to operate the technology, and aspects of the technology or method that make it user-friendly or user-unfriendly.

## Section 4 — Reference Laboratory Analyses

### Reference Laboratory Selection

The verification process is based on the presence of a statistically validated data set against which the performance goals of the technology may be compared. The choice of an appropriate reference method and reference laboratory are critical to the success of the demonstration. To assess the performance of the explosives field analytical technologies, the data obtained from demonstration participants were compared to data obtained using conventional analytical methods. Selection of the reference laboratory was based on the experience of prospective laboratories with QA procedures, reporting requirements, and data quality parameters consistent with the goals of the program. Specialized Assays, Inc. (currently part of Test America, Inc.), of Nashville, Tennessee, was selected to perform the analyses based on ORNL's experience with laboratories capable of performing explosives analyses using EPA SW-846 Method 8330. ORNL reviewed Specialized Assays' record of laboratory validation performed by the U.S. Army Corps of Engineers (Omaha, Nebraska). EPA and ORNL decided that, based on the credibility of the Army Corps program and ORNL's prior experience with the laboratory, Specialized Assays would be selected to perform the reference analyses.

ORNL conducted an audit of Specialized Assays' laboratory operations on May 4, 1999. This evaluation focused specifically on the procedures that would be used for the analysis of the demonstration samples. Results from this audit indicated that Specialized Assays was proficient in several areas, including quality management, document/record control, sample control, and information management. Specialized Assays was found to be compliant with implementation of Method 8330 analytical procedures. The company provided a copy of its QA plan, which details all of the QA and quality control (QC) procedures for all laboratory operations (Specialized Assays 1999). The audit team noted that Specialized Assays had excellent procedures in place for data backup, retrievability, and long-term storage. ORNL conducted a second audit at Specialized Assays

while the analyses were being performed. Since the initial qualification visit, management of this laboratory had changed because Specialized Assays became part of Test America. The visit included tours of the laboratory, interviews with key personnel, and review of data packages. Overall, no major deviations from procedures were observed and laboratory practices appeared to meet the QA requirements of the technology demonstration plan (ORNL 1999).

### Reference Laboratory Method

The reference laboratory's analytical method, presented in the technology demonstration plan, followed the guidelines established in EPA SW-846 Method 8330 (EPA 1994). According to Specialized Assays' procedures, soil samples were prepared by extracting 2-g samples of soil in acetonitrile by sonication for approximately 16 h. An aliquot of the extract was then combined with a calcium chloride solution to precipitate out suspended particulates. After the solution was filtered, the filtrate was ready for analysis. For the water samples, 400 mL of sample were combined with sodium chloride and acetonitrile in a separatory funnel. After mixing and allowing the solutions to separate, the bottom aqueous layer was discarded and the organic layer was collected. The acetonitrile volume was reduced to 2 mL, and the sample was diluted with 2 mL of distilled water for a final volume of 4 mL. The sample was then ready for analysis. The analytes were identified and quantified using a high-performance liquid chromatograph (HPLC) with a 254-nm UV detector. The primary analytical column was a C-18 reversed-phase column with confirmation by a secondary cyano column. The practical quantitation limits were 0.5 µg/L for water and 0.5 mg/kg for soils.

### Reference Laboratory Performance

ORNL validated all of the reference laboratory data according to the procedure described in the demonstration plan (ORNL 1999). During the validation, the following aspects of the data were reviewed: completeness of the data package, adherence to holding time requirements, correctness

of the data, correlation between replicate sample results, evaluation of QC sample results, and evaluation of spiked sample results. Each of these categories is described in detail in the demonstration plan. The reference laboratory reported valid results for all samples, so completeness was 100%. Preanalytical holding time requirements for water (7 days to extract; 40 days to analyze) and soil (14 days to extract; 40 days to analyze) were met. A few errors were found in a small portion of the data (~4%). Those data were corrected for transcription

and calculation errors that were identified during the validation. One data point, a replicate Iowa soil sample, was identified as suspect. The result for this sample was 0.8 mg/kg; the results from the other three replicates averaged 27,400 mg/kg. Inclusion or exclusion of this data point in the evaluation of comparability with the field technology (reported in Section 5) did not significantly change the *r* value, so it was included in the analysis. The reference laboratory results for QC samples were

**Table 3.** Summary of the Reference Laboratory Performance for Soil Samples

Statistic	Accuracy (% recovery)		Precision <sup>a</sup> (% RSD)			
	RDX N = 20	TNT N = 20	DNT <sup>b</sup> N <sub>R</sub> = 3 <sup>c</sup>	HMX N <sub>R</sub> = 13	RDX N <sub>R</sub> = 13	TNT N <sub>R</sub> = 18
Mean	102	100	56	29	25	29
Median	99	96	32	30	21	25
Range	84–141	76–174	14–123	12–63	4–63	2–72

<sup>a</sup>Calculated from those samples where all four replicates were reported as a detect.

<sup>b</sup>DNT represents total concentration of 2,4-DNT and 2,6-DNT.

<sup>c</sup>N<sub>R</sub> represents the number of replicate sets; N represents the number of individual samples

**Table 4.** Summary of the Reference Laboratory Performance for Water Samples

Statistic	Accuracy (% recovery)		Precision <sup>a</sup> (% RSD)			
	RDX N = 20	TNT N = 20	DNT <sup>b</sup> N <sub>R</sub> = 7 <sup>c</sup>	HMX N <sub>R</sub> = 20	RDX N <sub>R</sub> = 29	TNT N <sub>R</sub> = 28
Mean	91	91	30	20	22	24
Median	87	91	30	17	17	20
Range	65–160	66–136	8–80	6–49	5–66	5–86

<sup>a</sup>Calculated from those samples where all four replicates were reported as a detect.

<sup>b</sup>DNT represents total concentration of 2,4-DNT and 2,6-DNT.

<sup>c</sup>N<sub>R</sub> represents the number of replicate sets; N represents the number of individual samples

flagged when the results were outside the QC acceptance limits. The reference laboratory results were evaluated by a statistical analysis of the data. Due to the limited results reported for the other Method 8330 analytes, only the results for the major constituents in the samples (total DNT, TNT, RDX, and HMX) are evaluated in this report.

The accuracy and precision of the reference laboratory results for soil and water are summarized in Tables 3 and 4, respectively. Accuracy was assessed using the spiked samples, while precision was assessed using the results from both spiked and environmental samples. The reference laboratory results were unbiased (accurate) for both soil and water, as mean percentage recovery values were near 100%. The

reference laboratory results were precise; all but one of the mean RSDs were less than 30%. The one mean RSD that was greater than 30% (soil, DNT, 56%) was for a limited data set of three.

Table 5 presents the laboratory results for blank samples. A false positive result is identified as any detected result on a known blank. The concentrations of the false positive water results were low (<2 µg/L). For the soil samples, one false positive detection appeared to be a preparation error because the concentration was near 70,000 mg/kg. Overall, it was concluded that the reference laboratory results were unbiased, precise, and acceptable for comparison with the field analytical technology.

**Table 5.** Summary of the Reference Laboratory Performance on Blank Samples

Statistic	Soil				Water			
	DNT	HMX	RDX	TNT	DNT	HMX	RDX	TNT
Number of data points	20	20	20	20	20	20	20	20
Number of detects	0	0	0	2	1	0	2	4
% of fp results	0	0	0	10	5	0	10	20

## Section 5 — Technology Evaluation

### Objective and Approach

The purpose of this section is to present a statistical evaluation of the FAST 2000 data and determine the instrument's ability to measure explosives-contaminated water samples. RI elected not to analyze the soil samples described in Section 3. The technology's precision and accuracy are presented for RDX and TNT. Performance was evaluated on separate FAST 2000 systems: that is, one FAST 2000 instrument was employed to determine RDX concentrations, while a second was used to determine TNT concentrations. Differences in performance levels between the two analytes could be due either to differences in analyte properties or to differences between the two instruments. In addition, an evaluation of comparability through a one-to-one comparison with the reference laboratory data is presented. Other aspects of the technology (such as cost, sample throughput, hazardous waste generation, and logistical operation) are also evaluated in this section. The Appendix contains the raw data provided by the vendor that were used to assess the performance of the FAST 2000.

### Precision

Precision is the reproducibility of measurements under a given set of conditions. Precision was determined by examining the results of blind analyses for four replicate samples. Data were evaluated only for those samples where all four replicates were reported as a detection. For example, for RDX,  $N_R = 22$  represents a total of 88 sample analyses (22 sets of four replicates). A summary of the overall precision of the FAST 2000 for the water sample results is presented in Table 6. The mean RSDs were 52% and 76% for RDX and TNT, respectively, indicating that the water analyses were imprecise.

### Accuracy

Accuracy represents the closeness of the FAST 2000's measured concentrations to the known content of spiked samples. A summary of the FAST 2000's overall accuracy is presented in

Table 7. For the water samples, the mean recoveries for RDX and TNT were 192% and 316%

**Table 6.** Summary of the FAST 2000 Precision for Water Samples

Statistic	RSD <sup>a</sup> (%)	
	RDX $N_R = 22^b$	TNT $N_R = 12$
Mean	52	76
Median	46	80
Range	8–142	36–143

<sup>a</sup>Calculated from only those samples where all four replicates were reported as a detect.

<sup>b</sup> $N_R$  represents the number of replicate sets

**Table 7.** Summary of the FAST 2000 Accuracy for Water Samples

Statistic	Recovery (%)	
	RDX $N = 20$	TNT $N = 19$
Mean	192	316
Median	168	197
Range	81–580	82–1,110

316%, respectively. This indicated that the FAST 2000's performance for the spiked samples was biased high because the mean recoveries (and the medians) were greater than 100%.

### False Positive/False Negative Results

Table 8 shows the FAST 2000's performance for the 20 blank samples. RI reported the presence of RDX in four samples (24% fp results) and TNT in 16 samples (80% fp results). Note that the RDX data are

evaluated for only 17 of the 20 blank water samples. For RDX, RI reported three of the blanks as “ME” (matrix effects), indicating that the FAST 2000 could not generate a result because of matrix interferences.

**Table 8.** Summary of FAST 2000 False Positives on Blank Water Samples

Statistic	RDX	TNT
Number of data points	17	20
Number of detects	4	16
% of fp results	24%	80%
Number reported as “ME”	3	0

Table 9 summarizes the FAST 2000’s fp and fn results for all spiked and environmental samples by comparing the FAST 2000 result with the reference laboratory result. (See Section 3 for a more detailed discussion of this evaluation.) For the water samples, 2% of the RDX results and 16% of the TNT results were reported as false positives relative to the reference laboratory results (i.e., the laboratory reported the analyte as a nondetect and RI reported it as a detect). A small fraction of the samples (3% for each analyte) were reported as nondetects by RI (i.e., false negatives) for samples where the laboratory reported a detect.

### Completeness

Completeness is defined as the percentage of the 176 results that are judged to be useable (i.e., the result was not rejected). These results where RI reported “ME” (31 for RDX and 37 for TNT) are considered incomplete. Therefore, completeness was 82% for RDX and 79% for TNT.

### Comparability

Comparability refers to how well the field technology and reference laboratory data agreed. A one-to-one sample comparison of the FAST 2000 results and the reference laboratory results was performed for all spiked and environmental samples that were reported above the reporting limits. In Table 10, the comparability of the water

**Table 9.** Summary of the FAST 2000 Detect/Nondetect Performance Relative to the Reference Laboratory Results

Statistic	RDX	TNT
Number of data points <sup>a</sup>	128	119
Number of fp results	2	19
% of fp results	2%	16%
Number of fn results	4	4
% of fn results	3%	3%
Number reported as “ME”	28	37

<sup>a</sup> Excludes those values reported as “ME.”

results are presented in terms of correlation coefficients ( $r$ ) and slopes ( $m$ ). The  $r$  value for the comparison of the entire data set of TNT results was 0.23 ( $m = 1.81$ ). As shown in Table 10, if comparability is assessed for specific concentration ranges, the  $r$  value does not change dramatically for TNT. Depending on the concentration ranges selected, the  $r$  value ranged from 0.14 to 0.21.

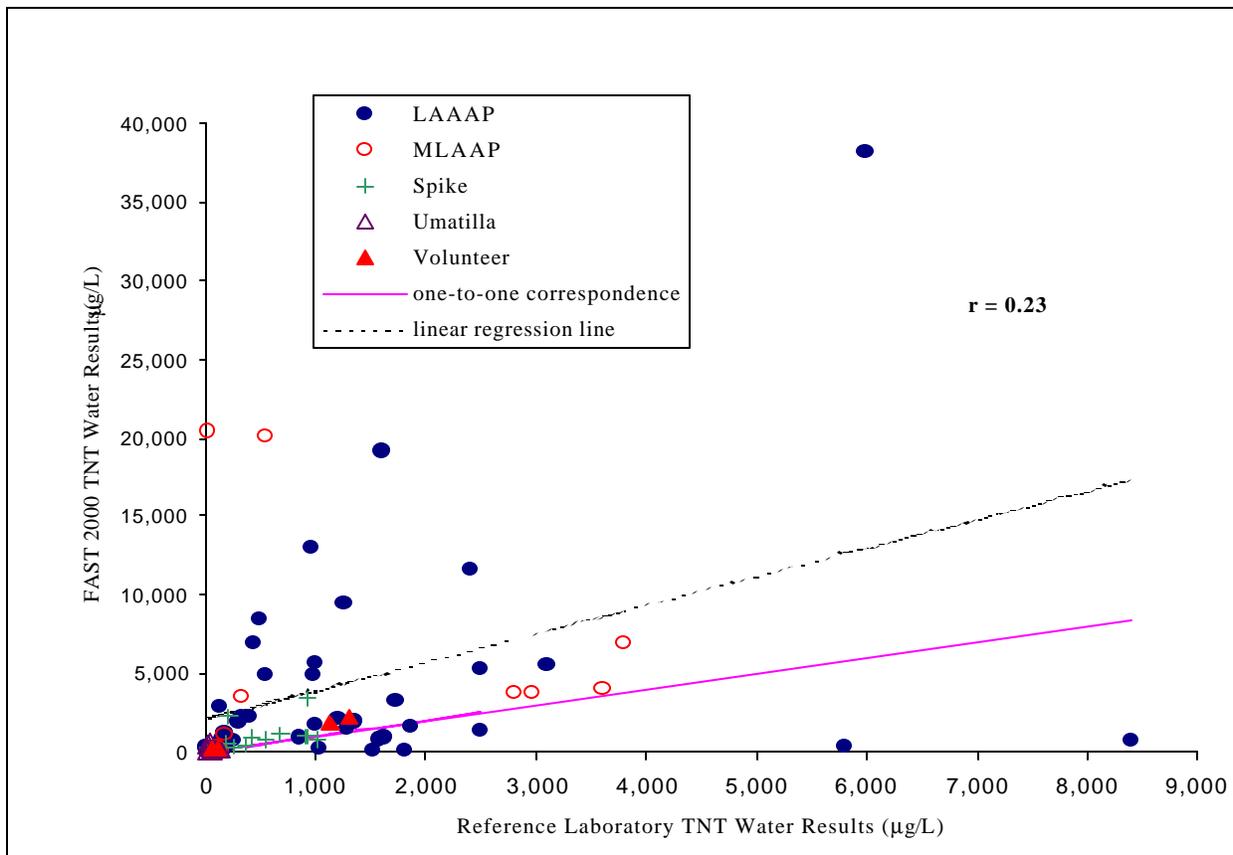
**Table 10.** FAST 2000 Correlation With Reference Data for Various Vendor Water Concentration Ranges

Concentration range	RDX		TNT	
	$r$	$m$	$r$	$m$
All values <sup>a</sup>	0.63	1.60	0.23	1.81
≤500 µg/L <sup>b</sup>	0.39	0.08	0.14	0.01
>500 µg/L	0.60	1.58	0.16	1.49
>1,000 µg/L	0.50	1.35	0.21	2.77

<sup>a</sup> Excludes those values reported as “< reporting limits.”

<sup>b</sup> Based on RI’s reported values.

A plot of the FAST 2000 results versus the reference laboratory results for all TNT concentrations is presented in Figure 2. The solid line on the graph represents an ideal one-to-one correspondence between the two measurements, while the dashed line

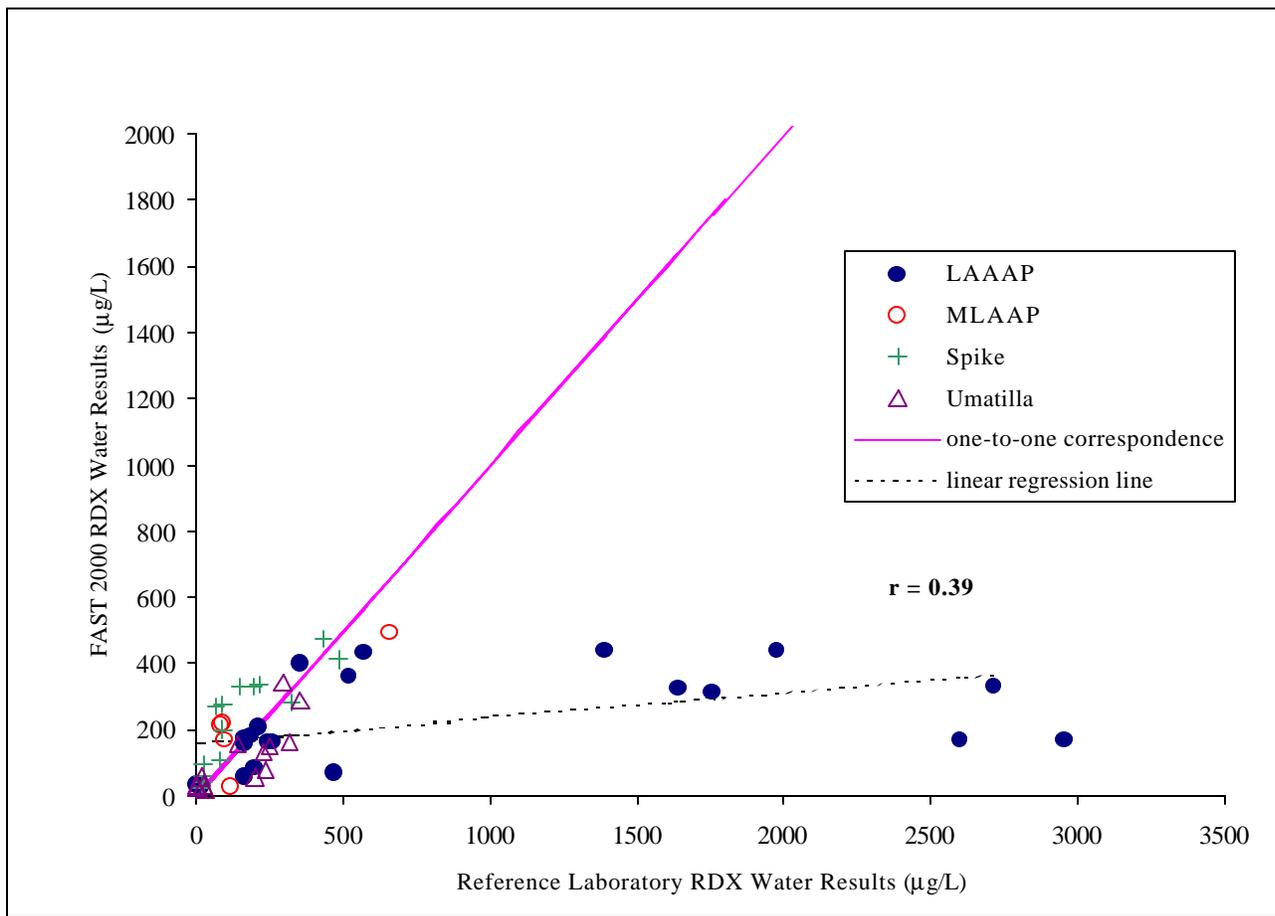


**Figure 2.** Comparability of reference laboratory water results with FAST 2000 results for all TNT concentrations. The slope of the linear regression line is 1.81 and the intercept is 2,135 µg/L. For clarity, one outlying MLAAP data point that is included in the regression analysis was excluded from the graph.

is the linear regression line. Overall, the FAST 2000's TNT results were generally higher than those of the reference laboratory, as indicated by the fact that the majority of the data points are above the solid line. For RDX, the correlation of the FAST 2000 results with the reference laboratory results was higher than for TNT, with a calculated  $r$  value of 0.63 and  $m$  of 1.60. Figure 3, a plot of the RDX comparability data for concentrations less than 500 µg/L, shows an interesting trend that further elaborates on the accuracy data previously presented. While the accuracy results were biased high for RDX spiked into distilled water, Figure 3 indicates that several of the FAST 2000 data were lower than the reference laboratory data. Further investigation of these data showed that the majority of the RDX results on the LAAAP samples were lower than the reference laboratory's matching results. The FAST 2000 results were generally higher for the spiked and MLAAP samples, and evenly

distributed higher and lower than the reference laboratory results for the Umatilla samples. This evaluation, summarized in Table 11, suggests a matrix-dependent effect. It should be noted, however, that the largest number of samples were analyzed from LAAAP; it is not known whether a similar trend would be observed with the samples from the other sites had more samples been analyzed. The evaluation of the TNT sample data by matrix concurred with the conclusion presented in the accuracy section, that the TNT results were generally biased high.

Another metric of comparability is the percent difference (%D) between the reference laboratory and the FAST 2000 results. The ranges of %D values for TNT and RDX are presented in Figure 4. Acceptable %D values would be between -25% and 25%, or near the middle of the  $x$ -axis of the plot. For TNT, the %D values were mostly greater than 75%. For RDX, the %D values were distributed among the



**Figure 3.** Comparability of reference laboratory water results with FAST 2000 results for vendor RDX concentrations less than 500 µg/L. The slope of the linear regression line is 0.08 and the intercept is 162 µg/L.

**Table 11.** Comparison of FAST 2000 and Reference Laboratory Results by Matrix

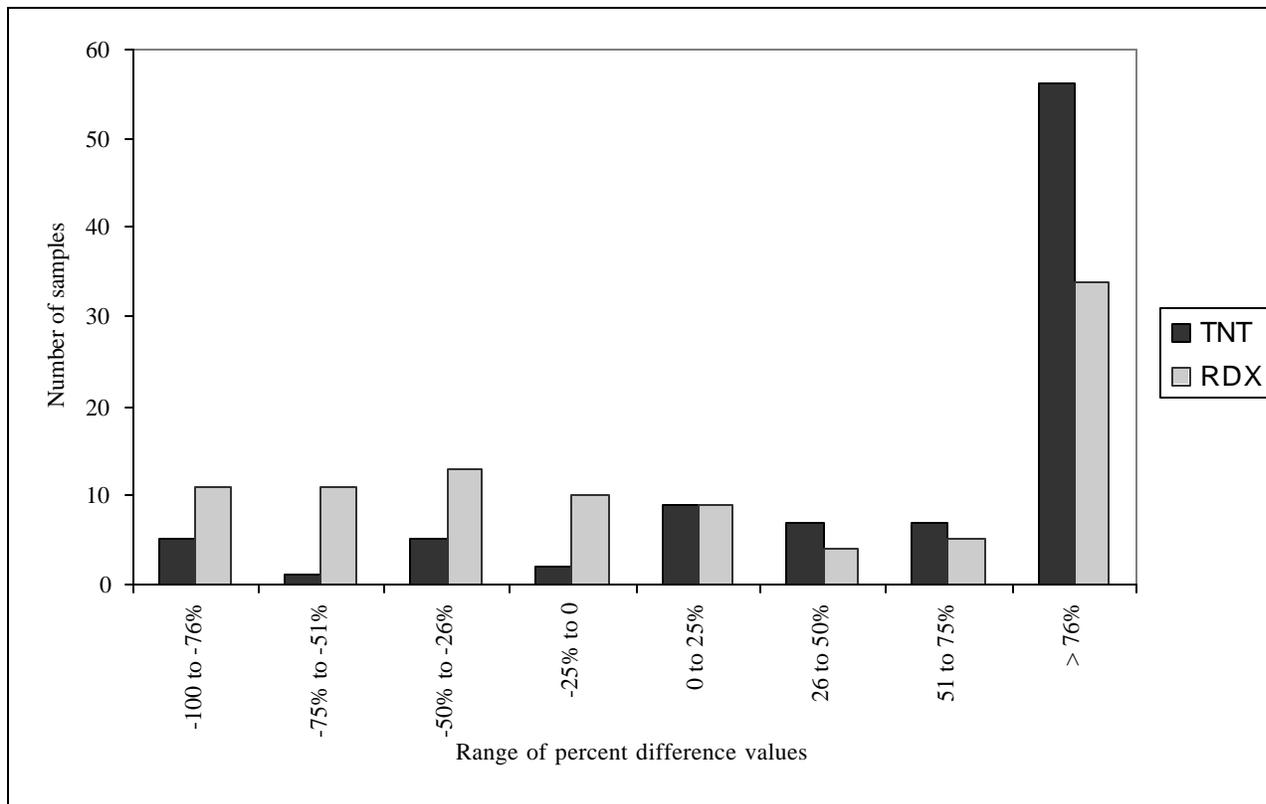
Sample or source type	RDX				TNT			
	N <sup>a</sup>	r <sup>b</sup>	m <sup>c</sup>	Comparison to reference laboratory <sup>d</sup>	N <sup>a</sup>	r <sup>b</sup>	m <sup>c</sup>	Comparison to reference laboratory <sup>d</sup>
Spiked	20	0.46	1.59	High	19	0.59	1.39	High
LAAAP	51	0.61	1.61	Low	42	0.36	1.41	High
MLAAP	13	0.56	0.89	High	15	0.11	1.84	High
Umatilla	13	0.80	0.63	Low and High	12	-0.32	-1.30	High
Volunteer	0	n/a	n/a	n/a	4	0.99	1.63	High

<sup>a</sup> Number of samples, excluding those reported as “ME” or as a nondetect.

<sup>b</sup> Correlation coefficient; FAST 2000 results versus reference laboratory results.

<sup>c</sup> Slope of linear regression line; FAST 2000 results versus reference laboratory results.

<sup>d</sup> Represents the majority of the measurements compared to the reference laboratory results.



**Figure 4.** Range of percent difference values for RDX and TNT.

various ranges, with the greatest number of samples having %D values greater than 75%. This supports the conclusion that the FAST 2000 RDX results were generally higher than those of the reference laboratory.

### **Sample Throughput**

Sample throughput is representative of the estimated amount of time required to prepare and analyze the sample. Operating under the outdoor conditions, the RI/ORNL team, usually consisting of three operators, accomplished a sample throughput rate of approximately three samples per hour for the water analyses. Separate instruments were used for the TNT and RDX analyses. Typically, two operators analyzed samples while one operator performed data analysis.

### **Ease of Use**

Three operators were typically used for the demonstration because of the number of demonstration samples and working conditions, but

the technology can be operated by a single person. Approximately one day of training would be necessary to operate the FAST 2000. RI offers training at its facility or at the user's facility. No particular level of educational training is required for the operator, but technician-level skills in chemical techniques would be advantageous.

### **Cost Assessment**

The purpose of this economic analysis is to estimate of the range of costs for an analysis of explosives-contaminated water samples using the FAST 2000 and a conventional analytical reference laboratory method. The analysis was based on the results and experience gained from this demonstration, costs provided by RI, and representative costs provided by the reference analytical laboratories that offered to analyze these samples. To account for the variability in cost data and assumptions, the economic analysis is presented as a list of cost elements and a range of costs for sample analysis by the FAST 2000 instrument and by the reference laboratory.

Several factors affected the cost of analysis. Where possible, these factors were addressed so that decision makers can complete a site-specific economic analysis to suit their needs. The following categories are considered in the estimate:

- sample shipment costs,
- labor costs,
- equipment costs, and
- waste disposal costs.

Each of these cost factors is defined and discussed and serves as the basis for the estimated cost ranges presented in Table 12. This analysis assumed that the individuals performing the analyses were fully trained to operate the technology. Costs for sample acquisition and pre-analytical sample preparation, which are tasks common to both methods, were not included here.

### **FAST 2000 Costs**

The costs associated with using the FAST 2000 included labor, equipment, and waste disposal costs. No sample shipment charges were associated with the cost of operating the FAST 2000 instrument because the samples were analyzed on-site.

#### **Labor**

Labor costs included mobilization/demobilization, travel, per diem expenses and on-site labor.

- *Mobilization/demobilization.* This cost element included the time for one person to prepare for and travel to each site. This estimate ranged from 5 to 8 h, at a rate of \$50/h.
- *Travel.* This element was the cost for the analyst(s) to travel to the site. If the analyst is located near the site, the cost of commuting to the site (estimated to be 50 miles at \$0.30/mile) would be minimal (\$15). The estimated cost of an analyst traveling to the site for this demonstration (\$1000) included the cost of airline travel and rental car fees.
- *Per diem expenses.* This cost element included food, lodging, and incidental expenses. The estimate ranged from zero (for a local site) to \$150/day for each analyst.
- *Rate.* The cost of the on-site labor was estimated at a rate of \$30–75/h, depending on

the required expertise level of the analyst. This cost element included the labor involved with the entire analytical process, comprising sample preparation, sample management, analysis, and reporting.

#### **Equipment**

Equipment costs included mobilization/demobilization, rental fees or purchase of equipment, and the reagents and other consumable supplies necessary to complete the analysis.

- *Mobilization/demobilization.* This included the cost of shipping the equipment to the test site. If the site is local, the cost would be zero. For this demonstration, the cost of shipping equipment and supplies was estimated at \$460.
- *Instrument purchase.* At the time of the demonstration, the cost of purchasing the FAST 2000 was \$23,650. The instrument purchase included a FAST 2000 instrument designed for use with an immunoassay-based sensor; a data acquisition card and a cable linking the instrument to the laptop computer; a fluid storage unit; one assay coupon kit; the software required to run the instrument and analyze data; and instruction manual. This price does not include the cost of the laptop computer. The instrument can be leased for \$1970 per month.
- *Reagents/supplies.* These items are consumable and are purchased on a per sample basis. At the time of the demonstration, the cost of the reagents and supplies needed to prepare and analyze water samples using the FAST 2000 was \$43 per sample. This cost included the sample preparation supplies, assay supplies, and consumable reagents. An ampule of standard was also available for approximately \$22.

#### **Waste Disposal**

Waste disposal costs are based on the 1999 regulations for disposal of explosives-contaminated waste. The analyses performed using the FAST 2000 instrument generated approximately 18 L of aqueous waste. ORNL's cost to dispose of the explosives-contaminated aqueous waste at a commercial facility was estimated at \$165 per 55-gal drum (the size that was used to contain this amount

of aqueous waste). There are mostly likely additional costs for labor associated with the waste disposal, but those costs are not estimated here.

### **Reference Laboratory Costs**

#### **Sample Shipment**

Sample shipment costs to the reference laboratory included the overnight shipping charges, as well as labor charges associated with the various organizations involved in the shipping process.

- *Labor.* This cost element included all of the tasks associated with the shipment of the samples to the reference laboratory. Tasks included packing the shipping coolers, completing the chain-of-custody documentation, and completing the shipping forms. The estimate to complete this task ranged from 2 to 4 h at \$50 per hour.
- *Overnight shipping.* The overnight express shipping service cost was estimated to be \$50 for one 50-lb cooler of samples.

#### **Labor, Equipment, and Waste Disposal**

The labor bids from commercial analytical reference laboratories that offered to perform the reference analysis for this demonstration ranged from \$150 to \$188 per sample. The bid was dependent on many factors, including the perceived difficulty of the sample matrix, the current workload of the laboratory, and the competitiveness of the market. This rate was a fully loaded analytical cost that included equipment, labor, waste disposal, and report preparation.

#### **Cost Assessment Summary**

An overall cost estimate for use of the FAST 2000 instrument versus use of the reference laboratory was not made because of the extent of variation in the different cost factors, as outlined in Table 12. The overall costs for the application of each technology will be based on the number of samples requiring analysis, the sample type, and the site location and characteristics. Decision-making

factors, such as turnaround time for results, must also be weighed against the cost estimate to determine the value of the field technology's providing immediate answers versus the reference laboratory's provision of reporting data within 30 days of receipt of samples.

#### **Miscellaneous Factors**

The following are general observations regarding the field operation and performance of the FAST 2000 instrument:

- The system, which weighed approximately 3 lb, was easily transportable.
- The RI/NRL team completely disassembled their work station at the close of each day. It took the team less than an hour each morning to prepare for sample analyses.
- The FAST 2000 was interfaced to the notebook computer through a PCMCIA card, through which both data and power connections were made. RI claimed that the instrument could run exclusively off of the computer's battery power. During the demonstration, the team found that the instrument worked best when the battery was removed and the computer was plugged into an electrical outlet.
- Sample preparation was minimal.
- Each sample was analyzed in duplicate, with the average concentration reported as the result. At least two calibration standards were analyzed with each sample.
- To analyze the 176 water samples, the RI/NRL team used 33 TNT-labeled membranes and 18 RDX-labeled membranes, averaging 5 samples per membrane for TNT and 10 samples per membrane for RDX.
- Data processing was performed using an NRL-written software program rather than with the data acquisition software package supplied with the instrument. The results were dependent on the user's designation of the start and the end of the analyte peak.

**Table 12.** Estimated Analytical Costs for Explosives-Contaminated Samples

<b>Analysis method:</b> FAST 2000		<b>Analysis method:</b> EPA SW-486 Method 8330	
<b>Analyst/manufacturer:</b> Research International		<b>Analyst/manufacturer:</b> Reference laboratory	
<b>Sample throughput:</b> 3 samples/h (for water)		<b>Typical turnaround:</b> 21 working days	
<b>Cost category</b>	<b>Cost (\$)</b>	<b>Cost category</b>	<b>Cost (\$)</b>
<b>Sample shipment</b>	0	<b>Sample shipment</b>	
		Labor	100–200
		Overnight shipping	50–150
<b>Labor</b>		<b>Labor</b>	
Mobilization/demobilization	250–400	Mobilization/demobilization	Included <sup>a</sup>
Travel	15–1,000 per analyst	Travel	Included
Per diem expenses	0–150/day per analyst	Per diem expenses	Included
Rate	30–75/h per analyst	Rate	150–188 per sample
<b>Equipment</b>		<b>Equipment</b>	Included
Mobilization/demobilization	0–460		
Instrument purchase price	23,650		
Instrument lease price	1,970 per month		
Reagents/supplies	43 per sample		
<b>Waste disposal</b>	165	<b>Waste disposal</b>	Included

<sup>a</sup> “Included” indicates that the cost is included in the labor rate.

### Summary of Performance

A summary of performance is presented in Table 13. Precision, defined as the mean RSD, was 52% and 76% for RDX and TNT water sample results, respectively. Accuracy, defined as the mean percent recovery relative to the spiked concentration, was 192% and 316% for RDX and TNT, respectively. Approximately 80% of the water analyses were complete; 20% were reported as “matrix effects,” where a result could not be determined. Comparison with Method 8330 results for homogeneous replicate splits indicated that the TNT results were generally higher than the reference laboratory results, while the RDX results were usually higher, but depended on the matrix analyzed. Of the 20 blank water samples, RI reported RDX in 4 samples (24% fp) and TNT in 16 samples (80% fp). False positive and false negative results were also determined by comparing the FAST 2000 result to the reference laboratory result on environmental and spiked samples. For RDX, 2% of the results were fp relative to the reference laboratory result, while 16% of the TNT results were reported as false positives. RI reported a small fraction of the samples (3% for

each analyte) as nondetects (i.e., false negatives) when the laboratory reported a detect.

The demonstration found that the FAST 2000 was relatively simple for the trained analyst to operate in the field, requiring less than an hour for initial setup. The sample throughput of the FAST 2000 was approximately three samples per hour. Three operators analyzed samples during the demonstration, but the technology can be run by a single trained operator. The overall performance of the FAST 2000 for the analysis of water samples was characterized as imprecise and biased high for TNT, and imprecise and biased high (but matrix-dependent) for RDX.

**Table 13.** Performance Summary for the FAST 20000 Water Analyses

<b>Feature/parameter</b>	<b>Performance summary</b>												
Precision	<b>Mean RSD</b> RDX: 52% TNT: 76%												
Accuracy	<b>Mean recovery</b> RDX: 192% TNT: 316%												
False positive results on blank samples	RDX: 24% TNT: 80%												
False positive results relative to reference laboratory results	RDX: 2% TNT: 16%												
False negative results relative to reference laboratory results	RDX: 3% TNT: 3%												
Comparison with reference laboratory results	<table> <tr> <td><b><i>r</i> (all results)</b></td> <td><b><i>m</i> (all results)</b></td> </tr> <tr> <td>RDX: 0.63</td> <td>1.60</td> </tr> <tr> <td>TNT: 0.23</td> <td>1.81</td> </tr> <tr> <td><b>Median %D</b></td> <td><b>Range of %D values</b></td> </tr> <tr> <td>RDX: 10%</td> <td>-94% to 8,167%</td> </tr> <tr> <td>TNT: 125%</td> <td>-96% to 157,000%</td> </tr> </table>	<b><i>r</i> (all results)</b>	<b><i>m</i> (all results)</b>	RDX: 0.63	1.60	TNT: 0.23	1.81	<b>Median %D</b>	<b>Range of %D values</b>	RDX: 10%	-94% to 8,167%	TNT: 125%	-96% to 157,000%
<b><i>r</i> (all results)</b>	<b><i>m</i> (all results)</b>												
RDX: 0.63	1.60												
TNT: 0.23	1.81												
<b>Median %D</b>	<b>Range of %D values</b>												
RDX: 10%	-94% to 8,167%												
TNT: 125%	-96% to 157,000%												
Completeness	RDX: 82% TNT: 79%												
Weight	2.8 lb												
Sample throughput	3 samples/h (three operators)												
Power requirements	Connect to portable PC (use battery or electrical power)												
Training requirements	One day instrument-specific training												
Cost	Instrument purchase: \$23,650 Instrument monthly lease: \$1,970 Supplies per sample: \$43												
Hazardous waste generation	18 L aqueous waste for 176 samples												
Overall evaluation	RDX: biased high (but matrix-dependent); imprecise TNT: biased high; imprecise												

## Section 6 — Technology Update and Representative Applications

*In this section, the vendor (with minimal editorial changes by ORNL) provides information regarding new developments with its technology since the verification activities. In addition, the vendor provides a list of representative applications in which its technology has been used.*

### Technology Update

As an outcome of the EPA trials, a decision has been made to improve the software algorithms used to quantify the assay data. After the trials it was discovered that approximately 10% of the errors in the FAST data were due to user error in analyzing the data. This was purportedly due to operator fatigue, after running assays for 10 hours a day over the period of several days. To avoid this problem in the future, the FAST software will be modified so that the assay quantification process is automated, eliminating the possibility of user error in postprocessing of the data.

In addition to the software improvements mentioned above, the Flow Assay Sensing and Testing system (FAST 2000) has now been replaced with a second-generation instrument, the FAST 6000 (shown in Figure 5). Research International has developed the FAST 6000 under contract to the U.S. Naval Research Laboratory (NRL). Unlike the FAST 2000, which requires connection to a laptop computer to run, the FAST 6000 is capable of stand-alone operation, running on a rechargeable lithium-ion battery pack. The instrument can be purchased in either a single-channel or a six-channel configuration. The form factor for a single-channel



Figure 5. The FAST 6000.

coupon is the same as that of a six-channel coupon, allowing single-channel FAST 6000 systems to be upgraded to six-channel systems at a later time if the user so desires. The six-channel instrument will significantly improve sample throughput and reduce the time needed for analysis of multiple analytes.

The FAST 6000 has a built-in 386 computer and  $4 \times 16$  character LCD display. A computer-controlled pump and valves completely automate the assay process for the user. Assay results are displayed on the LCD display, and the assay data is saved in an internal 2-MB FLASH disk. Assay data files taken into the field can be downloaded to a desktop computer at a later time via an RS-232 link. An advanced Windows-based software program has been developed to allow the user to transfer assay data and recipe files between an IBM-compatible personal computer and the FAST 6000. Files can be saved to the computer hard disk for later viewing and analysis. After a recipe is optimized, it can be transferred to the FAST 6000 for use in the field. This is useful for new assay development, in which timing and flow rates are being optimized by modifying easy to use assay recipes.

The software provides a real-time display of the data from the FAST 6000 in both a table format and a graphical format. The Windows-based software program also allows the user to run the FAST 6000 from a remote computer. With the addition of an optional RF data link, the FAST 6000 system can be run from a remote computer up to 20 miles away from the FAST 6000. This technology also makes it possible to create an array of systems that are operated and monitored by a single central computer.

The FAST 6000 has been improved considerably through the addition of a positive displacement pump and redesigned electronics with higher signal-to-

noise ratios. The ability to simultaneously run multiple assays for different analytes on a single system also represents a significant improvement. Research International continues to develop and improve the FAST technology. Work is under way on a six-channel version of the FAST that will be used to detect explosives underwater. This system is being designed to automatically sample salt water at depths up to 300 feet and run assays for TNT and RDX.

### **Representative Applications**

**1995:** Prototypes of the laboratory version of the continuous flow immunosensor participated in field demonstrations with funding from the Strategic Environmental Research and Development Program (SERDP). It was tested at Umatilla Army Depot in Hermiston, Oregon, and SUBASE Bangor in Bangor, Washington, in collaboration with U.S. EPA Region 10. Results for the continuous flow immunosensor can be found in several refereed papers (see below). EPA coordinator Harry Craig and associates have written a report of the field trials and has a proceedings paper describing both sensors.

**1997:** At the National Environmental Technology Test Site (NETTS) at the Volunteer Army Ammunition Plant in Chattanooga, Tennessee, an on-site demonstration of FAST 2000 was conducted September 23–27. Groundwater was tested for TNT in samples from 10 monitoring wells during a 4-day trial. The demonstration was conducted as part of a SERDP research program. Also, Harry Craig, EPA Region 10, purchased two FAST 2000 units for monitoring at SUBASE Bangor and Umatilla Army Depot.

**1997–1998:** Three field trials for groundwater analysis and one for soil were performed using the FAST 2000 to perform on-site analysis for validation studies. The first groundwater test for this project was conducted June 23–27, 1997 at

SUBASE Bangor, Bangor, Washington The second site was Umatilla Army Depot in Hermiston, Oregon, where the second field trial took place August 4–8, 1997. The third field trial site was the Naval Surface Weapons Center in Crane, Indiana,

where groundwater tests were performed September 8–12, 1997. The soil field trial was held April 27– May 1, 1998 at Manchester, Washington, on samples from Umatilla Army Depot. Harry Craig, EPA Region 10, coordinated the sites and sample collection and provided non-developer operators for these trials.

### **Refereed Papers**

Bart, J. C., L. L. Jidd, K. E. Hoffman, A. M. Wilkins, P. T. Charles, and A. W. Kusterbeck. 1997. "Application of a Portable Immunosensor to Detect Explosives TNT and RDX in Groundwater Samples." *Environmental Science and Technology* 31(5): 1505–11.

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Craig, H., G. Furguson, A. Markos, A. Kusterbeck, L. Shriver-Lake, T. Jenkins, and P. Thorne. 1996. "Field Demonstration of On-Site Analytical Methods for TNT and RDX in Groundwater." Pp. 204–19 in *Proceedings of the Great Plains–Rocky Mountain Hazardous Substance Research Center (HSRC)/ Waste Education and Research Consortium (WERC) Joint Conference on the Environment*, May 21–23, Albuquerque, N.M.

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ORNL (Oak Ridge National Laboratory). 1999. "Technology Demonstration Plan: Evaluation of Explosives Field Analytical Techniques." Oak Ridge National Laboratory, Oak Ridge, Tenn., August.

Specialized Assays, Inc. 1999. "Comprehensive Quality Assurance Plan." SAL-QC-Rec 5.0. January 6.

## Appendix

### RI's FAST 2000 Sample Results Compared with Reference Laboratory Results

Sample site or type	Sample no.	Sample replicate	RDX <sup>a</sup> (mg/L)		TNT <sup>a</sup> (mg/L)		RI analysis order <sup>b</sup>
			RI	Ref Lab	RI	Ref Lab	
Blank	6	1	<20.0	1.5	46	1.9	2068
Blank	6	2	<20.0	<0.5	65	1.2	2069
Blank	6	3	<20.0	<0.5	<20.0	<0.5	2005
Blank	6	4	<20.0	<0.5	226	<0.5	2112
Blank	7	1	<20.0	<0.5	97	<0.5	2142
Blank	7	2	ME <sup>c</sup>	<0.5	124	0.9	2065
Blank	7	3	22	<0.5	160	<0.5	2038
Blank	7	4	<20.0	<0.5	<20.0	<0.5	2012
Blank	8	1	81	<0.5	116	<0.5	2045
Blank	8	2	131	<0.5	172	<0.5	2044
Blank	8	3	ME	0.5	196	<0.5	2027
Blank	8	4	60	<0.5	194	1.3	2050
Blank	9	1	<20.0	<0.5	<20.0	<0.5	2025
Blank	9	2	<20.0	<0.5	120	<0.5	2078
Blank	9	3	<20.0	<0.5	112	<0.5	2165
Blank	9	4	<20.0	<0.5	112	<0.5	2146
Blank	10	1	<20.0	<0.5	48	<0.5	2176
Blank	10	2	ME	<0.5	81	<0.5	2034
Blank	10	3	<20.0	<0.5	<20.0	<0.5	2042
Blank	10	4	<20.0	<0.5	46	<0.5	2004
Louisiana	5	1	84	200	1280	170	2123
Louisiana	5	2	58	159	2862	136	2104
Louisiana	5	3	181	180	757	151	2049
Louisiana	5	4	153	158	311	138	2033
Louisiana	6	1	203	210	213	162	2031
Louisiana	6	2	162	238	207	176	2163
Louisiana	6	3	176	160	ME	113	2094
Louisiana	6	4	162	256	273	178	2159
Louisiana	7	1	ME	<0.5	ME	0.5	2130
Louisiana	7	2	ME	<0.5	59	<0.5	2026

Sample site or type	Sample no.	Sample replicate	RDX <sup>a</sup> ( $\mu\text{g/L}$ )		TNT <sup>a</sup> ( $\mu\text{g/L}$ )		RI analysis order <sup>b</sup>
			RI	Ref Lab	RI	Ref Lab	
Louisiana	7	3	ME	<0.5	ME	<0.5	2139
Louisiana	7	4	20	<0.5	25	<0.5	2039
Louisiana	8	1	ME	<0.5	<20.0	<0.5	2003
Louisiana	8	2	<20.0	1.1	217	0.5	2103
Louisiana	8	3	ME	<0.5	ME	<0.5	2010
Louisiana	8	4	ME	<0.5	ME	<0.5	2160
Louisiana	9	1	894	1760	1986	300	2153
Louisiana	9	2	434	1390	700	240	2052
Louisiana	9	3	1300	1410	2331	320	2157
Louisiana	9	4	323	1640	ME	330	2134
Louisiana	10	1	1099	560	ME	65	2119
Louisiana	10	2	67	470	ME	40	2150
Louisiana	10	3	364	520	<20.0	30	2116
Louisiana	10	4	906	256	138	28	2035
Louisiana	11	1	<20.0	18.6	106	<0.5	2121
Louisiana	11	2	<20.0	17.2	ME	<0.5	2066
Louisiana	11	3	<20.0	13	ME	<0.5	2147
Louisiana	11	4	28	13.9	250	<0.5	2030
Louisiana	12	1	ME	89	ME	101	2096
Louisiana	12	2	<20.0	34	ME	59	2155
Louisiana	12	3	ME	52	184	134	2089
Louisiana	12	4	<20.0	104	ME	131	2076
Louisiana	13	1	<20.0	2.5	287	1.1	2154
Louisiana	13	2	<20.0	1.8	20	<0.5	2014
Louisiana	13	3	<20.0	2	ME	<0.5	2156
Louisiana	13	4	<20.0	2.3	179	<0.5	2141
Louisiana	14	1	<20.0	11.8	1396	<0.5	2082
Louisiana	14	2	<20.0	11.4	ME	<0.5	2086
Louisiana	14	3	<20.0	14	367	<0.5	2120
Louisiana	14	4	26	14	ME	<0.5	2067
Louisiana	15	1	ME	<0.5	ME	<0.5	2013
Louisiana	15	2	ME	<0.5	ME	<0.5	2070
Louisiana	15	3	ME	<0.5	ME	<0.5	2124

Sample site or type	Sample no.	Sample replicate	RDX <sup>a</sup> ( $\mu\text{g/L}$ )		TNT <sup>a</sup> ( $\mu\text{g/L}$ )		RI analysis order <sup>b</sup>
			RI	Ref Lab	RI	Ref Lab	
Louisiana	15	4	ME	<0.5	700	<0.5	2028
Louisiana	16	1	<20.0	<0.5	<20.0	<0.5	2118
Louisiana	16	2	<20.0	<0.5	1005	<0.5	2115
Louisiana	16	3	<20.0	<0.5	715	<0.5	2168
Louisiana	16	4	29	0.8	ME	0.7	2058
Louisiana	17	1	514	2160	1899	1360	2102
Louisiana	17	2	328	2720	925	1620	2006
Louisiana	17	3	170	2600	832	1580	2032
Louisiana	17	4	312	1760	ME	1240	2093
Louisiana	18	1	3367	19600	ME	6900	2151
Louisiana	18	2	31270	16700	358	5800	2040
Louisiana	18	3	55700	22800	664	8400	2062
Louisiana	18	4	16097	18400	38230	6000	2114
Louisiana	19	1	73070	6100	5582	3100	2085
Louisiana	19	2	5330	3100	19114	1600	2173
Louisiana	19	3	7260	3500	11674	2400	2143
Louisiana	19	4	7684	4900	1322	2500	2110
Louisiana	20	1	430	570	3310	1720	2001
Louisiana	20	2	398	350	12960	950	2113
Louisiana	20	3	589	380	5666	990	2164
Louisiana	20	4	526	315	4864	985	2172
Louisiana	21	1	863	940	6904	440	2161
Louisiana	21	2	756	1180	8467	490	2057
Louisiana	21	3	561	1410	4860	540	2088
Louisiana	21	4	701	1130	2283	400	2075
Louisiana	22	1	3942	3780	1501	1280	2002
Louisiana	22	2	166	2960	ME	1080	2135
Louisiana	22	3	702	2780	2220	1210	2117
Louisiana	22	4	6891	2680	1739	1000	2071
Louisiana	23	1	2924	2340	59	1520	2041
Louisiana	23	2	564	1430	917	850	2009
Louisiana	23	3	991	1710	221	1040	2011
Louisiana	23	4	887	1930	ME	1260	2072

Sample site or type	Sample no.	Sample replicate	RDX <sup>a</sup> ( $\mu\text{g/L}$ )		TNT <sup>a</sup> ( $\mu\text{g/L}$ )		RI analysis order <sup>b</sup>
			RI	Ref Lab	RI	Ref Lab	
Louisiana	24	1	583	1770	9440	1260	2097
Louisiana	24	2	672	3000	5260	2500	2162
Louisiana	24	3	730	2260	1637	1860	2109
Louisiana	24	4	434	1980	102	1810	2083
Milan	6	1	744	9	110	80	2149
Milan	6	2	756	235	292	100	2175
Milan	6	3	582	250	375	105	2023
Milan	6	4	657	170	258	60	2101
Milan	7	1	1622	670	3984	3600	2127
Milan	7	2	492	660	6936	3800	2063
Milan	7	3	826	580	3807	2960	2140
Milan	7	4	549	650	ME	2650	2128
Milan	8	1	ME	<50.0	3536	320	2132
Milan	8	2	55	<50.0	96893	1610	2091
Milan	8	3	24	120	20080	540	2043
Milan	8	4	ME	<50.0	3770	2800	2131
Milan	9	1	ME	36	2130	<10.0	2136
Milan	9	2	ME	<10.0	28546	<10.0	2137
Milan	9	3	ME	19	20450	13	2166
Milan	9	4	<20.0	<10.0	7370	<10.0	2051
Milan	10	1	166	93	72	154	2111
Milan	10	2	220	91	169	149	2079
Milan	10	3	210	84	<20.0	150	2107
Milan	10	4	503	96	1193	167	2133
Spike/PE	7	1	106	83	116	19.8	2020
Spike/PE	7	2	275	88	257	22	2074
Spike/PE	7	3	197	88	ME	20.5	2092
Spike/PE	7	4	271	65.5	168	17.4	2036
Spike/PE	8	1	22	17	316	72	2148
Spike/PE	8	2	91	19	225	77	2126
Spike/PE	8	3	56	22	145	90.5	2169
Spike/PE	8	4	44	19	140	66	2015

Sample site or type	Sample no.	Sample replicate	RDX <sup>a</sup> ( $\mu\text{g/L}$ )		TNT <sup>a</sup> ( $\mu\text{g/L}$ )		RI analysis order <sup>b</sup>
			RI	Ref Lab	RI	Ref Lab	
Spike/PE	9	1	<20.0	<0.5	553	185	2108
Spike/PE	9	2	<20.0	42	394	244	2019
Spike/PE	9	3	<20.0	<0.5	300	185	2105
Spike/PE	9	4	<20.0	<0.5	2224	212	2122
Spike/PE	10	1	332	188	157	<0.5	2047
Spike/PE	10	2	282	320	<20.0	1.1	2167
Spike/PE	10	3	331	146	121	<0.5	2048
Spike/PE	10	4	338	210	195	<0.5	2099
Spike/PE	11	1	5802	650	432	350	2087
Spike/PE	11	2	810	1480	1139	680	2138
Spike/PE	11	3	2045	840	800	550	2029
Spike/PE	11	4	1640	810	883	420	2018
Spike/PE	12	1	896	460	1000	930	2080
Spike/PE	12	2	410	480	818	1020	2059
Spike/PE	12	3	476	430	3451	930	2007
Spike/PE	12	4	521	470	1068	910	2016
Umatilla	1	1	77	234	103	42	2021
Umatilla	1	2	57	200	712	34	2144
Umatilla	1	3	130	228	ME	32	2054
Umatilla	1	4	157	142	ME	20	2060
Umatilla	2	1	ME	<0.5	ME	<0.5	2170
Umatilla	2	2	<20.0	<0.5	323	0.6	2100
Umatilla	2	3	30	2.6	36	1.3	2037
Umatilla	2	4	ME	1.5	ME	1.3	2064
Umatilla	3	1	20	27	108	146	2008
Umatilla	3	2	31	23	276	117	2073
Umatilla	3	3	<20.0	20	<20.0	109	2098
Umatilla	3	4	<20.0	27	145	127	2084
Umatilla	4	1	ME	15	ME	57	2125
Umatilla	4	2	<20.0	4.8	220	27	2056
Umatilla	4	3	58	12	51	83	2061
Umatilla	4	4	ME	15	244	96	2171
Umatilla	5	1	289	348	405	<0.5	2077
Umatilla	5	2	343	296	ME	0.5	2158
Umatilla	5	3	159	316	<20.0	<0.5	2090
Umatilla	5	4	150	248	325	<0.5	2152

Sample site or type	Sample no.	Sample replicate	RDX <sup>a</sup> ( $\mu\text{g/L}$ )		TNT <sup>a</sup> ( $\mu\text{g/L}$ )		RI analysis order <sup>b</sup>
			RI	Ref Lab	RI	Ref Lab	
Umatilla	6	1	ME	5.1	ME	28	2129
Umatilla	6	2	ME	3.5	226	22.5	2106
Umatilla	6	3	25	3.3	605	12.3	2046
Umatilla	6	4	ME	5.9	<20.0	20.8	2145
Volunteer	4	1	<20.0	<0.5	ME	54	2055
Volunteer	4	2	ME	<0.5	ME	44.5	2017
Volunteer	4	3	ME	<0.5	224	63	2081
Volunteer	4	4	<20.0	1.8	231	105	2022
Volunteer	5	1	<20.0	<5.0	ME	840	2053
Volunteer	5	2	ME	<5.0	2276	1290	2174
Volunteer	5	3	<20.0	<5.0	1812	1130	2024
Volunteer	5	4	ME	<50.0	ME	890	2095

<sup>a</sup> The data are presented exactly as reported. Note that the data are not consistently reported with the same number of significant figures.

<sup>b</sup> These are the sample numbers from which the analysis order can be discerned. For example, 2001 was analyzed first, then 2002, etc.

<sup>c</sup> “ME” indicates that the sample contained matrix effects and the result could not be reported by the FAST 2000.